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NEWS	3	APR 03	CAS coverage of exemplified prophetic substances enhanced
NEWS	4	APR 07	STN is raising the limits on saved answers
NEWS	5	APR 24	CA/CAPLUS now has more comprehensive patent assignee information
NEWS	6	APR 26	USPATFULL and USPAT2 enhanced with patent assignment/reassignment information
NEWS	7	APR 28	CAS patent authority coverage expanded
NEWS	8	APR 28	ENCOMPLIT/ENCOMPLIT2 search fields enhanced
NEWS	9	APR 28	Limits doubled for structure searching in CAS REGISTRY
NEWS	10	MAY 08	STN Express, Version 8.4, now available
NEWS	11	MAY 11	STN on the Web enhanced
NEWS	12	MAY 11	BEILSTEIN substance information now available on STN Easy
NEWS	13	MAY 14	DGENE, PCTGEN and USGENE enhanced with increased limits for exact sequence match searches and introduction of free HIT display format
NEWS	14	MAY 15	INPADOCDB and INPAFAMDB enhanced with Chinese legal status data
NEWS	15	MAY 28	CAS databases on STN enhanced with NANO super role in records back to 1992
NEWS	16	JUN 01	CAS REGISTRY Source of Registration (SR) searching enhanced on STN
NEWS	17	JUN 26	NUTRACEUT and PHARMAML no longer updated
NEWS	18	JUN 29	IMSCOPROFILE now reloaded monthly
NEWS	19	JUN 29	EPFULL adds Simultaneous Left and Right Truncation (SLART) to AB, MCLM, and TI fields
NEWS	20	JUL 09	PATDPAFULL adds Simultaneous Left and Right Truncation (SLART) to AB, CLM, MCLM, and TI fields
NEWS	21	JUL 14	USGENE enhances coverage of patent sequence location (PSL) data
NEWS	22	JUL 14	CA/CAPLUS to be enhanced with new citing references features
NEWS	23	JUL 16	GBFULL adds patent backfile data to 1855
NEWS EXPRESS	MAY 26 09	CURRENT WINDOWS VERSION IS V8.4, AND CURRENT DISCOVER FILE IS DATED 06 APRIL 2009.	

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	ENTRY	SESSION
FULL ESTIMATED COST	0.22	0.22

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DICTIONARY FILE UPDATES: 17 JUL 2009 HIGHEST RN 1164451-20-0

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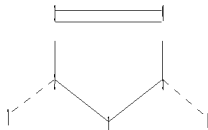
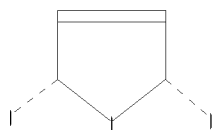
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chain nodes :  
6 7

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ring nodes :  
1 2 3 4 5  
chain bonds :  
2-7 5-6  
ring bonds :  
1-2 1-5 2-3 3-4 4-5  
exact/norm bonds :  
2-7 5-6  
exact bonds :  
1-2 1-5 2-3 3-4 4-5  
isolated ring systems :  
containing 1 :

Match level :  
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:CLASS 7:CLASS

L1 STRUCTURE UPLOADED

=> s l1

SAMPLE SEARCH INITIATED 11:12:52 FILE 'REGISTRY'  
SAMPLE SCREEN SEARCH COMPLETED - 8532 TO ITERATE

23.4% PROCESSED 2000 ITERATIONS 50 ANSWERS  
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)  
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*  
BATCH \*\*COMPLETE\*\*  
PROJECTED ITERATIONS: 165103 TO 176177  
PROJECTED ANSWERS: 45344 TO 51238

L2 50 SEA SSS SAM L1

=> s l1 full

FULL SEARCH INITIATED 11:13:01 FILE 'REGISTRY'  
FULL SCREEN SEARCH COMPLETED - 172473 TO ITERATE

100.0% PROCESSED 172473 ITERATIONS 45136 ANSWERS  
SEARCH TIME: 00.00.01

L3 45136 SEA SSS FUL L1

=> file caplus

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	185.88	186.10

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FILE COVERS 1907 - 20 Jul 2009 VOL 151 ISS 4  
FILE LAST UPDATED: 19 Jul 2009 (20090719/ED)  
REVISED CLASS FIELDS (/NCL) LAST RELOADED: Jun 2009  
USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Jun 2009

CAPLUS now includes complete International Patent Classification (IPC) reclassification data for the second quarter of 2009.

CAS Information Use Policies apply and are available at:

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This file contains CAS Registry Numbers for easy and accurate substance identification.

The ALL, BIB, MAX, and STD display formats in the CA/CAPLUS family of databases will soon be updated to include new citing references information. This enhancement may impact record import into database management software. For additional information, refer to NEWS 22.

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=> s l3 and pyrrolidine
      83153 L3
      33258 PYRROLIDINE
      3141 PYRROLIDINES
      34301 PYRROLIDINE
          (PYRROLIDINE OR PYRROLIDINES)
L4      294 L3 AND PYRROLIDINE
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          31 OXOS
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          (OXO OR OXOS)
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      3141 PYRROLIDINES
      34301 PYRROLIDINE
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      1054 OXO(L)PYRROLIDINE
L5      17 L4 AND (OXO(L)PYRROLIDINE)
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      846073 CATALYSTS
      1089292 CATALYST
          (CATALYST OR CATALYSTS)
L6      2 L5 AND CATALYST
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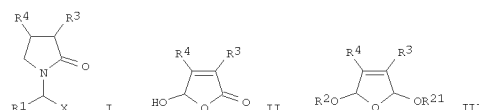
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L6 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2009 ACS on STN  
ACCESSION NUMBER: 2005:283464 CAPLUS  
DOCUMENT NUMBER: 142:336241  
TITLE: Process for preparing N-substituted 2-oxo-  
pyrrolidines such as levetiracetam from  
dihydrofurans and amines.  
INVENTOR(S): Surtees, John; Lurquin, Francoise; Diouf, Ousmane  
PATENT ASSIGNEE(S): Ucb, S.A., Belg.  
SOURCE: PCT Int. Appl., 19 pp.  
CODEN: PIXXD2  
DOCUMENT TYPE: Patent  
LANGUAGE: English  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

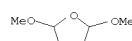
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005028435	A1	20050331	WO 2004-EP10212	20040913
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
AU 2004274157	A1	20050331	AU 2004-274157	20040913
CA 2538938	A1	20050331	CA 2004-2538938	20040913
EP 1667967	A1	20060614	EP 2004-765134	20040913
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, FI, RO, CY, TR, BG, CZ, EE, HU, PL, SK				
BR 2004014436	A	20061114	BR 2004-14436	20040913
CN 1882535	A	20061220	CN 2004-80033998	20040913
ZA 2006001970	A	20070530	ZA 2006-1970	20040913
JP 2007515387	T	20070614	JP 2006-527306	20040913
IN 2006DN01133	A	20070817	IN 2006-DN1133	20060303
MX 2006003024	A	20061110	MX 2006-3024	20060316
KR 2006121889	A	20061129	KR 2006-705793	20060323
US 20070100150	A1	20070503	US 2007-572579	20070103
PRIORITY APPLN. INFO.:			EP 2003-21534	A 20030924
			WO 2004-EP10212	W 20040913

OTHER SOURCE(S): CASREACT 142:336241; MARPAT 142:336241  
GI

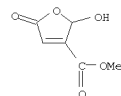
L6 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2009 ACS on STN (Continued)



AB Title compds. [I; R1 = Ra, Rb; R3, R4 = H, OH, SH, halo, cyano, CO2H, SO3H, Ra, Rb, etc.; X = CONR5R6, CO2R7, cyano; R5-R7 = H, Ra, Rb; Ra = (substituted) alkyl; Rb = (substituted) aryl, heteroaryl, heterocycloalkyl], were prepared by reaction of dihydrofurans (II, III; R2, R21 = alkyl, aralkyl) with R1CH(NH2)X in the presence of H2 and a catalyst. Thus, (S)-2-aminobutyramide and 2,5-dimethoxy-2,5-dihydrofuran were stirred 1.5 h in aqueous HCl; Na2CO3 was added to bring the pH to 8-9. Pd/C in H2O/EtOH and H2 were added to give 13% levetiracetam.  
IT 332-77-4, 2,5-Dimethoxy-2,5-dihydrofuran 96695-26-0  
RI: RCT (Reactant); RACT (Reactant or reagent)  
(preparation of substituted oxopyrrolidines such as levetiracetam from dihydrofurans and amines)  
RN 332-77-4 CAPLUS  
CN Furan, 2,5-dihydro-2,5-dimethoxy- (CA INDEX NAME)

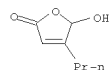


RN 96695-26-0 CAPLUS  
CN 3-Furanocarboxylic acid, 2,5-dihydro-2-hydroxy-5-oxo-, methyl ester (CA INDEX NAME)



IT 78920-10-2P  
RI: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(preparation of substituted oxopyrrolidines such as levetiracetam from

L6 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2009 ACS on STN (Continued)  
dihydrofurans and amines)  
RN 78920-10-2 CAPLUS  
CN 2(5H)-Furanone, 5-hydroxy-4-propyl- (CA INDEX NAME)



REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE  
FORMAT

L6 ANSWER 2 OF 2 CAPLUS COPYRIGHT 2009 ACS on STN  
ACCESSION NUMBER: 1956:89209 CAPLUS  
DOCUMENT NUMBER: 50:89209  
ORIGINAL REFERENCE NO.: 50:16777d-1,16778a-1,16779a-i,16780a-i,16781a-i,16782a-i,16783a-b  
TITLE: Ethynylation. V. Reactions of hydrated ethynylation products. Dehydration of  $\gamma$ -alkanediols  
AUTHOR(S): Reppe, Walter; et al.  
SOURCE: Annalen der Chemie, Justus Liebig's (1955), 596, 80-158  
CODEN: 9X224Y  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable  
OTHER SOURCE(S): CASREACT 50:89209  
AB XIIa is prepared in quant. yield from 1 kg. (HOCH2CH2)2 stirred and heated to 165° with 100 g. 80% H3PO4, with fresh IX added and the temperature raised slowly to 185° as product distilled, and the distillate dried over K2CO3 and redistd., b. 65-7°, d20 0.8886, nD20 1.4065; azeotrope containing 5.5% H2O b. 66°. XIIa is also prepared from IX with p-MeC6H4SO3H, (CO2H)2, (CH2CO2H)2, maleic anhydride, or resorcinol-(H3O3)2C6H3CHOCH2O resin as catalyst, by heating 35% aqueous IX with H3PO4 and NH4Cl in an autoclave to 280° 10 h., or from 25 g. IX passed over 325 g. precipitated and calcined Cr2O3 at 300-25° or CaHPO4 at 300°. Dehydration of the corresponding R1R2C(OH)CH2CH2C(OH)R3R4 gives the following cyclic ethers (R1, R2, R3, R4, and catalyst given): Me, H, H, H (XXXV), H3PO4-H2SO4, 78-9°; Me, H, Me, H, K2S2O7, 91.5° (azeotrope containing 13% H2O, b. 78°), forms with ferrocyanic acid an H2O-insol., colorless, crystalline adduct; Me, Me, Me, Me, H3PO4 or K2S2O7, b. 114-17°. 1,1'-Ethylenebicyclohexanol (120 g.) gives, with 2 mL. 25% H2SO4 in vacuo at 150°, 85 g. 2,2',5,5'-bis(pentamethylene)-XIIa, b2 93-5°. (CH2)2CH2 is prepared by passing 20-5 g. IX and 4-5 g. H2O/h. at 300-50° over 300 mL. catalyst prepared by mixing 100 g. anhydrous NaH2PO4 with 40 mL. H2O, adding 8 g. BuH2PO4 and 20 g. graphite, evaporating with continuous stirring, heating finally to 160°, and breaking to suitable size. The same catalyst, with 98% H3PO4 instead of Bu phosphate, gave 90% (CH2)2CH2 from XIIa at 280°; 1800 g. XXXV gives 510 g. MeCH:CHCH:CH2, b. 40°, and 720 g. recovered XXXV. (MeCH(OH)CH2)2 (500 g./day), passed at 280-300° over catalyst prepared by adding 125 g. Al(OH)3 to 700 mL. H2O and 700 g. 90% H3PO4, stirring 2-3 h. at 110-15°, adding 185 g. NaH2PO4 and 43 g. BuNH2, cooling, adding 320 g. 34.4% water glass, ball milling, evaporating in vacuo at 260°, and crushing, gives 240 g. (MeCH:CH)2, b. 77-8°. 2,5-Dihydrofuran (17 g.), prepared from 50 g. XXV and 2 g. 20% H2SO4 at 125-30° and 18 mm., b. 63.5° (forms azeotrope containing 7.5% H2O). XXV is also dehydrated by (CO2H)2 at 170°, by passing in HCl at 140° or by passing it over Al2O3 at 240-50°. 2,5-di-Me-XXXVa, b. 90-3°, and 2,2',5,5'-bis(pentamethylene)dihydrofuran, b16 123-5°, are prepared similarly. 1,2,4-Butanetriol (300 g.) and 10 mL. concentrated H2SO4 at 30 mm. and 100-15° treated with an addnl. 1500 g. triol and distilled during 36 h. give 1350 g. 3-HO-XIIa (XXXVI), b740 183°, b20 93-5°; acetate, b12 64°. XXXVI (88 g.) added at -5° to 108 g. COCl2, warmed to 20° in 1 h., and swept with dry air, the residual

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L6 ANSWER 2 OF 2 CAPLUS COPYRIGHT 2009 ACS on STN (Continued)  
 chloroformate (XXXVII) treated with 100 mL. NH<sub>4</sub>OH at 20-40°, then a stream of NH<sub>3</sub> gas, and extd. with Et<sub>2</sub>O, and the ext. evapd. gives 100 g. XXXVI urethane (C<sub>5</sub>H<sub>9</sub>NO<sub>3</sub>), m. 91° (from C<sub>6</sub>H<sub>6</sub>). PhNH<sub>2</sub> and XXXVII give the N-Ph urethane, m. 112°, and H<sub>2</sub>N(CH<sub>2</sub>)<sub>6</sub>NH<sub>2</sub> gives N,N'-hexamethylenebis(tetrahydro-3-furyl urethane), m. 113°. XXXVI (176 g.), 1 g. Na, and 125 g. CH<sub>2</sub>CHCN give β-(tetrahydro-3-furyloxy)propionitrile, b<sub>2</sub> 108-10°. HOCH<sub>2</sub>CH<sub>2</sub>(CH(OH))<sub>2</sub>Me and concd. H<sub>2</sub>SO<sub>4</sub> at 150° give 1-methyl-4-hydroxy-XIIa, b. 183°, b<sub>2</sub> 90-1°. 2,3-dichloro-XIIa (XXXVIII) b<sub>2</sub> 62° (1200 g.) is prepd. by passing Cl into 1500 g. XIIa at 0-10° 60 h.; 500 g. 2-(4-chlorobutoxy)-3-chloro-XIIa (XXXIX), b<sub>2</sub> 145-55°, is also formed. XXXVIII (75%) and XXXIX (25%) are also prepd. by passing XIIa and Cl dild. with N over glass beads at 100°; XXXIX is prepd. in quant. yield by passing HCl into 156 g. XIIa and 280 g. XXXVIII at 20-30° (or in 330 g. yield from 200 g. XXXVIII and 180 g. Cl(CH<sub>2</sub>)<sub>4</sub>OH 2 h. at 100°). XXXVIII (1 g.) chlorinated several days at 100° gives 890 g. b<sub>2</sub> 7-30 120-4°, 1200 g. b<sub>2</sub> 140-2°, and hexachloro XIIa isolated from the mixt. m. 40°. XXXVIII (300 g.), warmed with 500 mL. H<sub>2</sub>O and 250 g. CaCO<sub>3</sub> gives 97 g. 2-hydroxy-3-chloro-XIIa (YOH) (XL), b<sub>2</sub> 92-5°, and 32 g. Y<sub>2</sub>O, b<sub>2</sub> 6 106°. The following ethers are prepd. from XXXVIII and ROH at 100°: YOMe, b. 150°, b<sub>2</sub> 50-1°; YOEt, b<sub>2</sub> 60-2°; YOBU, b<sub>2</sub> 86-8°; YOC<sub>12</sub>H<sub>25</sub>-n, b<sub>2</sub> 166-7°; YOC<sub>2</sub>H<sub>2</sub>CH<sub>2</sub>OH, b<sub>2</sub> 127-30°; (YOC<sub>2</sub>H<sub>2</sub>)<sub>2</sub>, b<sub>2</sub> 140-5°; the IX YOC<sub>2</sub>H<sub>2</sub>C.tpbond.CCH<sub>2</sub>OH, b<sub>2</sub> 5 135°; (YOC<sub>2</sub>H<sub>2</sub>C.tpbond.)<sub>2</sub>, b<sub>2</sub> 8 168-9°; YOCH(CH<sub>2</sub>OV)<sub>2</sub>, b<sub>2</sub> 4 205°; YOC<sub>2</sub>H<sub>2</sub>CH(OV)CH<sub>2</sub>CH<sub>2</sub>OV, b<sub>2</sub> 150-80°. Other YR prepd. from XXXVIII (R, yield, wt. XXXVIII, wt. other reagent, and conditions given): AcO (b<sub>2</sub> 5 110-15°), 200 g., 420 g., 181 g. AcOH, refluxed 1 h.; NaO<sub>3</sub>S, 380 g., 280 g., 500 g. cryst. Na<sub>2</sub>SO<sub>3</sub> in H<sub>2</sub>O; cyano (b<sub>2</sub> 1 97-9°), -, 140 g. and 120 g. CuCN at 100° [concd. H<sub>2</sub>SO<sub>4</sub> give the acid, m. 91° (Et ester, b<sub>2</sub> 100-6°)]; Ph (b<sub>2</sub> 10 123-32°), 154 g., 280 g. 1. C<sub>6</sub>H<sub>6</sub> treated several hrs. at 5-10° with BF<sub>3</sub>; 1-naphthyl (b<sub>2</sub> 5 171-3°), 1 l. C<sub>6</sub>H<sub>6</sub> treated several hrs. with AlCl<sub>3</sub>. XXXVIII (41 g.), and 76 g. (NH<sub>2</sub>)<sub>2</sub>CS in 400 mL. H<sub>2</sub>O refluxed 4 h. and neutralized with 1100 g. 40% NaOH give 110 g. 2-amino-5-(2-hydroxyethyl)thiazole, m. 98.5° (from C<sub>6</sub>H<sub>6</sub>); mono-HCl salt, m. 227°; mono-Ac deriv., m. 209°, di-Ac deriv., m. 163-4°. Urea gives a low yield of 2-amino-5-(2-hydroxyethyl)oxazole, m. 132-3° (from alc.); mono-HCl salt, m. 207° (from AcOH). XIIa satd. in the cold with HCl every 12 h. for 48 h., shaken with H<sub>2</sub>O, and neutralized, gives HO(CH<sub>2</sub>)<sub>4</sub>Cl, b<sub>2</sub> 76-8°, also prepd. from (CH<sub>2</sub>CH<sub>2</sub>OH)<sub>2</sub> and HCl at 80° with 5% BiCl<sub>3</sub>-SiO<sub>2</sub>. Cl(CH<sub>2</sub>)<sub>4</sub>Cl (XLI), b<sub>2</sub> 19 55-6° (560 g.), is prepd. from 360 g. XIIa, 10 g. anhyd. ZnCl<sub>2</sub>, and 650 g. SOCl<sub>2</sub> at 130°; it is also prepd. from XIIa and anhyd. HCl 5 h. at 150° or from XIIa, concd. H<sub>2</sub>SO<sub>4</sub>, and concd. HCl in 4 h. at 170°. XIIa (870 g.), 60 g. concd. H<sub>2</sub>SO<sub>4</sub>, and 750 g. SOCl<sub>2</sub> kept 15 h. at 65-85° and another 100 g. SOCl<sub>2</sub> added at 100-10° give 970 g. O[(CH<sub>2</sub>)<sub>4</sub>Cl]<sub>2</sub>, b<sub>2</sub> 125-8°. A similar reaction at 100°, 72 h. gives some [Cl(CH<sub>2</sub>)<sub>4</sub>OCCH<sub>2</sub>CH<sub>2</sub>]<sub>2</sub>, b<sub>2</sub> 6144-7°. The following compds. are prepd. in generally lower yield from the corresponding monosubstituted XIIa by reactions analogous to the foregoing: ClCH<sub>2</sub>CH(OCt)CH<sub>2</sub>CH<sub>2</sub>Cl, b<sub>2</sub>

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 under N 4 h. at 100-10°, then Raney Co and H at 80-100°, 200 atm.; PhNH(CH<sub>2</sub>)<sub>4</sub>CN, b<sub>2</sub> 17 155-65° (PhNH<sub>2</sub> overnight at 110°), this with Raney Co and H at 100°, 200 atm. gives PhNH(CH<sub>2</sub>)<sub>5</sub>NH<sub>2</sub>, b<sub>2</sub> 133-5°, o-MeC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>NH(CH<sub>2</sub>)<sub>4</sub>CN, b<sub>2</sub> 236-8° [procedure not given], and cyclobutyl cyanide, b. 149°, b<sub>2</sub> 55° (by refluxing with NaNH<sub>2</sub> in dry Et<sub>2</sub>O, this with 50% NaOH gives the acid, b. 190-1°). δ-Valerolactone (250 g.) and 100 g. NaCN heated to 230°, the melt cooled and dissolved in H<sub>2</sub>O after the reaction subsided, and the soln. decolorized with C, acidified, and extd. with Et<sub>2</sub>O gives 120 g. NC(CH<sub>2</sub>)<sub>4</sub>CO<sub>2</sub>H, b<sub>2</sub> 162-70°; the Et ester, b<sub>2</sub> 145-50°, gives with alc. NH<sub>3</sub>, Raney Co, and 200 atm. H at 130° e-caprolactam, m. 65°, b<sub>2</sub> 140-2°. XLII (720 g.), 720 g. dry NaCN, 30 g. CaCO<sub>3</sub>, and 5 g. NaHCO<sub>3</sub> heated to 160°, 800 g. XLI added so that the temp. remained at 180-200°, and the mixt. heated another 2 h. give 600 g. newly formed XLIII. NH<sub>2</sub>(CH<sub>2</sub>)<sub>5</sub>CN, b<sub>2</sub> 106° (Bz deriv., m. 96-7°), (200 g.) is prepd. from 440 g. XLIII, 130 g. NH<sub>3</sub>, and 30 g. Raney Ni under 20 atm. H at 130°; at 120-40° and 120-50 atm., the product is H<sub>2</sub>N(CH<sub>2</sub>)<sub>6</sub>NH<sub>2</sub>, b<sub>2</sub> 81-5°, m. 45-6° (N,N'-diformyl deriv., m. 112°; N,N'-di-Ac deriv., m. 127°). XLI (126 g.) added at 80-5° to 150 g. anhyd. NaCN in 340 g. H<sub>2</sub>O and 100 mL. alc. and stirred 18 h. at this temp. gives 44 g. tetrahydrothiophene, b. 119-22°. O[(CH<sub>2</sub>)<sub>4</sub>R]<sub>2</sub> prepd. from O[(CH<sub>2</sub>)<sub>4</sub>Cl]<sub>2</sub> are (R, constn., reagents, and conditions given in parentheses): OH, b<sub>2</sub> 15 150-5° (8% Na<sub>2</sub>CO<sub>3</sub> 2 h. at 150°) (diacetate, b<sub>2</sub> 139-40°); HCO<sub>2</sub>, b<sub>2</sub> 8 120-30° (aq.-alc. NaO<sub>2</sub>C 2 h. at 140°); SH, b<sub>2</sub> 6 113° (aq. Na<sub>2</sub>S, satd. with H<sub>2</sub>S, 10 h. at 145°); NH<sub>2</sub>, b<sub>2</sub> 1.4 105-8° (large excess NH<sub>3</sub>, alc., and alkali earth oxide a short time at 100-20°); cyano, b<sub>2</sub> 172-6° (aq.-alc. NaCN 5 h. at 130°) (hydrogenation gives O[(CH<sub>2</sub>)<sub>5</sub>NH]<sub>2</sub>, b<sub>2</sub> 137-8°). [HO(CH<sub>2</sub>)<sub>4</sub>]<sub>2</sub>O (720 g.), or (CH<sub>2</sub>CH<sub>2</sub>OH)<sub>2</sub>, and 240 g. NaOH heated to 180°, 800 g. O[(CH<sub>2</sub>)<sub>4</sub>Cl]<sub>2</sub> added, the mixt. stirred 0.5 h., distd. with H<sub>2</sub>O, neutralized, and the oil distd. to about 250°/0.5 mm. gives 700 g. residual dihydroxypolybutyl ether, HO no. 170; (CH<sub>2</sub>CH<sub>2</sub>OH)<sub>2</sub> and O[(CH<sub>2</sub>)<sub>4</sub>Cl]<sub>2</sub> give a similar product. Longer heating gives higher-mol.-wt. products. O[(CH<sub>2</sub>)<sub>4</sub>Cl]<sub>2</sub> in anhyd. C<sub>6</sub>H<sub>6</sub> treated with Na at 100-5° (cooling) gives H[(O(CH<sub>2</sub>)<sub>4</sub>)<sub>2</sub>OH] 50 g. of this, added to 200 g. 65% HNO<sub>3</sub> at 50-5° give H<sub>2</sub>O<sub>2</sub>(CH<sub>2</sub>)<sub>6</sub>CO<sub>2</sub>H, m. 138°. XLII (560 g.) treated during 6 h. with 390 g. Cl at 10-10° gives 664 g. CH<sub>2</sub>ClCHClCCl<sub>2</sub>Me, b. 203-6°, b<sub>2</sub> 92-3°, and 110 g. (CH<sub>2</sub>ClCHCl)<sub>2</sub> (XLV), m. 73° (from ligroine), b<sub>2</sub> 130-40°. XLII (250 g.) and 10 g. Bz<sub>2</sub>O<sub>2</sub> heated to 80°, 550 g. SO<sub>2</sub>Cl<sub>2</sub> added, and the mixt. warmed 3 h. at 80-100° give 140 g. XLV, 170 g. CH<sub>2</sub>ClCCl<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl, b<sub>2</sub> 86-8°, and 35 g. pentachlorobutane, b<sub>2</sub> 102°. (CHCH<sub>2</sub>OH)<sub>2</sub> prepd. by refluxing XLII and NaOH or KOH in ROH are (R given): Me, b<sub>2</sub> 141°; Et, b<sub>2</sub> 61-3°; iso-Pr, b<sub>2</sub> 80-2°; CH<sub>2</sub>CHCH<sub>2</sub>, b<sub>2</sub> 106°; Bu, b<sub>2</sub> 128°; Ph, b<sub>2</sub> 175° (decomp.); MeC<sub>6</sub>H<sub>4</sub>, b<sub>2</sub> 4 170-6°; cyclohexyl, b<sub>2</sub> 160°; PhCH<sub>2</sub>, b<sub>2</sub> 137-9°; tetrahydro-3-furyl, b<sub>2</sub> 148°; tetrahydrofurfuryl, b<sub>2</sub> 150°. The bis-MeOCH<sub>2</sub>CH<sub>2</sub> ether, b<sub>2</sub> 105-6°, and the bis-BuOCH<sub>2</sub>CH<sub>2</sub> ether, b<sub>2</sub> 148-50°, are prepd. similarly. XXV (500 g.) treated with 160 g. powd. NaOH (cooling), 70 mL. H<sub>2</sub>O distd. in vacuo, 250 g. XLII added slowly with stirring at 110-20° and the temp. held 2 h. gives viscous, nondistillable, H<sub>2</sub>O-sol. tri(butenediol), HO no. 480; diacetate, C<sub>16</sub>H<sub>24</sub>O<sub>6</sub>, b<sub>2</sub> 178-82°. XLII (500 g.) added to 40 g. XXV and 350 g. NaOH

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 65-80°, and 4,4'-dichlorodithoxydibutyl ether, b<sub>2</sub> 140-5°; ClCH<sub>2</sub>CH(OMe)CH<sub>2</sub>CH<sub>2</sub>Cl, b<sub>2</sub> 5 67°, and 4,4'-dichlorodimethoxydibutyl ether; Cl(CH<sub>2</sub>)<sub>3</sub> CHClMe, b<sub>2</sub> 53-4°, and (MeCHClCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl)<sub>2</sub>, b<sub>2</sub> 125-40°; ClCH<sub>2</sub>CHMeCH<sub>2</sub>CH<sub>2</sub>Cl, b<sub>2</sub> 8-9 46-66°, and 4,4'-dichlorodimethylidibutyl ether, b<sub>2</sub> 80 125-38°. Similarly XXI gives (MeCHClCH<sub>2</sub>)<sub>2</sub>, b. 170-2°. Tetrahydropyran (172 g.), 20 g. anhyd. ZnCl<sub>2</sub>, and 300 g. SOCl<sub>2</sub> refluxed 42 h. give 167 g. Cl(CH<sub>2</sub>)<sub>5</sub>Cl, b<sub>2</sub> 75-6°. XXXVa (280 g.), 15 g. anhyd. ZnCl<sub>2</sub>, and 540 g. SOCl<sub>2</sub> 7 h. at 80-5° give 100 g. (ClCH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub> (XLI), b<sub>2</sub> 148-51°, b. 145°, better prepd. (410-g. yield) from 320 g. XXV refluxed with an aq. eqmt. of (CH<sub>2</sub>Cl)<sub>2</sub> or C<sub>6</sub>H<sub>6</sub> in an HCl stream with azeotropic distn. of H<sub>2</sub>O; at 0°, mostly ClCH<sub>2</sub>CH:CHCH<sub>2</sub>OH, b<sub>2</sub> 10 75-6°, is formed. Substitution of HBr for HCl in the corresponding reaction gives Br(CH<sub>2</sub>)<sub>4</sub>Br, b<sub>2</sub> 90-3°; Br(CH<sub>2</sub>)<sub>5</sub>Br, b<sub>2</sub> 18 104-5°; and (BrCH<sub>2</sub>CH)<sub>2</sub>, b<sub>2</sub> 14 74-6°, m. 54°. XIIa (72 g.), 148 g. BuOH, and 13 g. concd. H<sub>2</sub>SO<sub>4</sub> heated 5 h. at 155° give 46 g. (BuOCH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>, b<sub>2</sub> 116-20°. XIIa (148 g.), contg. some Zn, treated with 158 g. AcCl gives 500 g. Cl(CH<sub>2</sub>)<sub>4</sub>OCt, b<sub>2</sub> 19 90-2°. Other Cl(CH<sub>2</sub>)<sub>4</sub>OCt prepd. analogously, using Zn or ZnCl<sub>2</sub> are (R and constn. given): ClCH<sub>2</sub>, b<sub>2</sub> 17 124°; Pr, b<sub>2</sub> 11 116-18°; Ph, b<sub>2</sub> 10 176-8°; p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, b<sub>2</sub> 0 167-72°, m. 38°; p-HSO<sub>3</sub>C<sub>6</sub>H<sub>4</sub>, b<sub>2</sub> 90-5°; Cl, b<sub>2</sub> 10 90° [Cl(CH<sub>2</sub>)<sub>4</sub>OCtNH<sub>2</sub>, m. 77° (from aq. alc.)]; [CH<sub>2</sub>NHCO<sub>2</sub>(CH<sub>2</sub>)<sub>4</sub>Cl]<sub>2</sub>, m. 104° (from MeOH); [CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NHCO<sub>2</sub>(CH<sub>2</sub>)<sub>4</sub>Cl]<sub>2</sub>, m. 94° (from MeOH). o-C<sub>6</sub>H<sub>4</sub>(COCl)<sub>2</sub> (203 g.), 144 g. XIIa, and Zn give 250 g. o-C<sub>6</sub>H<sub>4</sub>(CO<sub>2</sub>(CH<sub>2</sub>)<sub>4</sub>Cl)<sub>2</sub>. Similarly XXXVIII gives ClCH<sub>2</sub>CH<sub>2</sub>CHClCHClOAc, b<sub>2</sub> 50 154-8°, and XXXV gives MeCHCl(CH<sub>2</sub>)<sub>3</sub>OCt, b<sub>2</sub> 21 92-5°. XXXV (172 g.) and 230 g. Ac<sub>2</sub>O heated 12 h. at 240° in a Cu-lined autoclave give 110 g. AcOCHMe(CH<sub>2</sub>)<sub>3</sub>OCt, b<sub>2</sub> 22 123-6°, in a tube at 240° the product is pentenyl acetate, b. 149-50°, this is hydrolyzed to a 1:1 mixt. (shown by MnO<sub>4</sub> oxidn.) of 3- and 4-penten-1-ol, b. 140-1°. Tetrahydrofurfuryl alc., AcCl, Zn dust, and C<sub>6</sub>H<sub>6</sub> give a diacetate, b<sub>2</sub> 158-60°, either AcOCH<sub>2</sub>CH(OAc)(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>Cl or AcOCH<sub>2</sub>CHCl(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>OAc; XXXVa similarly gives ClCH<sub>2</sub>CH:CHCH<sub>2</sub>OAc, b<sub>2</sub> 92-4°. A mixt. of 3 mol HCN and 1 mol XIIa passed at 40 mL/h. over Al<sub>2</sub>O<sub>3</sub> at 425° gives product contg. (mol. %): 13 XIIa, 38 CH<sub>2</sub>CHCH<sub>2</sub>CH<sub>2</sub>CN, b. 140-2°, 26 C<sub>5</sub>H<sub>5</sub>N, and 3 neutral and 12 basic high boiling fractions. XLI (650 g.), 83 g. PCl<sub>3</sub>, and 17 g. red P chlorinated at 120-60° and irradiated with a 500-w. light gives CH<sub>2</sub>ClCHClCH<sub>2</sub>CH<sub>2</sub>Cl, b<sub>2</sub> 74°, CH<sub>2</sub>ClCClCH<sub>2</sub>CH<sub>2</sub>Cl, b<sub>2</sub> 86-8°, CH<sub>2</sub>ClCCl<sub>2</sub>CHClCH<sub>2</sub>Cl, b<sub>2</sub> 102°, (CH<sub>2</sub>ClCCl<sub>2</sub>)<sub>2</sub>, b<sub>2</sub> 115°, and CHCl<sub>2</sub>CCl<sub>2</sub>CCl<sub>2</sub>CH<sub>2</sub>Cl, b<sub>2</sub> 125°. PhOH (188 g.) and 83 g. NaOH heated to 150° and 127 g. XLI added give, after 1 h., 120 g. PhO(CH<sub>2</sub>)<sub>4</sub>OPh, m. 97-8° (from alc.). XLI (380 g.), 700 mL. BuOH, and 200 g. KCN at 100-20° give 140 g. unseparated XLI, 42 g. NC(CH<sub>2</sub>)<sub>4</sub>CN (XLIII), and 140 g. CH<sub>2</sub>Cl(CH<sub>2</sub>)<sub>3</sub>CN (XLIV), b<sub>2</sub> 16 100-3°. Comps. prepd. from XLIV are (reagents, time, and temp. in parentheses): δ-Valerolactone, b<sub>2</sub> 8 110-15° (hydrazide, C<sub>5</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>, m. 107°), and its polymer (H<sub>2</sub>O, 4 h., 180°); piperidine (NH<sub>3</sub>, Raney Co, and H, 6 h. at 70°, 100 atm.); piperidone together with N-4-cyanobutylpiperidone, b<sub>2</sub> 10 200-15° (NH<sub>3</sub> at 70-120° in an autoclave, low yield); Et<sub>2</sub>N(CH<sub>2</sub>)<sub>4</sub>CN, b<sub>2</sub> 45 130-5° (Et<sub>2</sub>NH 6-10 h. at 100-20°); Et<sub>2</sub>N(CH<sub>2</sub>)<sub>5</sub>NH<sub>2</sub>, b<sub>2</sub> 27 103-5° (Et<sub>2</sub>NH and Raney Co under N 6-8 h. at 120°, then H at 100 atm.); BuNH(CH<sub>2</sub>)<sub>5</sub>NH<sub>2</sub>, b<sub>2</sub> 10 165-8° (BuNH<sub>2</sub>

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 refluxing in 900 g. XIIa 2 h., the solvent distd., and the residue allowed to crystallize gives XXV cyclic diether, C<sub>8</sub>H<sub>12</sub>O<sub>2</sub>, m. 117°, b. 184°; hydrogenation over Pd at 120° gives the satd. cyclic diether, C<sub>8</sub>H<sub>16</sub>O<sub>2</sub>, b. 183-5°. (cCH<sub>2</sub>R)<sub>2</sub> prepd. from XLII were (R and reagents, and conditions in parentheses given): morpholino, b<sub>2</sub> 0.9-1 141-2° (morpholine 1 h. at 100°); 1-pyrrolidinyl, b<sub>2</sub> 0 98-105° (pyrrolidine several hrs. at 90-100°); 2-benzothiazolylthio, m. 86° (from alc.) (2-mercaptobenzothiazole and NaOH in XIIa); NaO<sub>3</sub>S, no constn. (aq. Na<sub>2</sub>SO<sub>3</sub> 7 h. on H<sub>2</sub>O bath); and CN, b<sub>2</sub> 14 150-80°, m. 76° (from alc.) (alc. NaCN refluxed 5 h.). The latter compd. decomp. somewhat when the crude is distd., even in the presence of methylene blue. XLI (500 g.) added with stirring at room temp. during 8 h. to 700 mL. C<sub>6</sub>H<sub>6</sub> and 80 g. anhyd. AlCl<sub>3</sub> and heated to 80° until HCl evolution ceases gives 10 g. tetrahydronaphthalene, b<sub>2</sub> 0.3 45°, 20 g. Ph(CH<sub>2</sub>)<sub>4</sub>Ph, b<sub>2</sub> 0.2 70-85°, 60 g. of a mixt. (b<sub>2</sub> 0.2 85-100°) of octahydroanthracene and -phenanthrene, 25 g. octahydro-anthracene, m. 70°, b<sub>2</sub> 100-30° (this with MnO<sub>4</sub> gives pyromellitic acid), and, from the residue, 68 g. dodecahydrotetraphenylene (XLVI). XLVI (460

g. from 950 g. XLI and 500 mL. C<sub>6</sub>H<sub>6</sub> treated with 500 g. anhyd. AlCl<sub>3</sub> during 48 h. (b<sub>2</sub> 140-20°), m. 239° (from PhCl), and fluoresces blue-white in UV light. Similarly prepd. in low yield are: from PhMe, a mixt., b<sub>2</sub> 140-5°, of 9-methyl-1,2,3,4,5,6,7,8-octahydroanthracene and 9-methyl-1,2,3,4,5,6,7,8-tetrahydronaphthalene; from m-C<sub>6</sub>H<sub>4</sub>Me<sub>2</sub>, dimethyltetrahydronaphthalene, b<sub>2</sub> 0.25 86-7° (dehydrogenation over ZnO gives dimethylnaphthalene; picrate, m. 118-19°), and C<sub>16</sub>H<sub>22</sub>, b<sub>2</sub> 0.3 156°, m. 132° (dehydrogenation gives anthracene and a hydrocarbon, m. 87°); from ClOH<sub>8</sub> a mixt. (b<sub>2</sub> 100-20°) of tetrahydronaphthalene and -phenanthrene and some (4-chlorobutyl)naphthalene, b<sub>2</sub> 140-50°; from Ph<sub>2</sub> a resin. m-C<sub>6</sub>H<sub>4</sub>Me<sub>2</sub> (212 g.), 230 g. CH<sub>2</sub>Br(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>Me, and 45 g. AlCl<sub>3</sub> at 90-100° give 130 g. of a mixt. (b<sub>2</sub> 130-130°) of (apparently) 1,5,7- and 4,5,7-trimethyltetrahydronaphthalene. C<sub>6</sub>H<sub>6</sub> (312 g.), 20 g. AlCl<sub>3</sub>, and 254 g. XLII give 32 g. hexahydrotetraphenylene. A polymeric oxo acid is prepd. from xylene, AlCl<sub>3</sub>, XLI, and phthalic anhydride. Naphthalene (1500 g.), 570 mL. XIIa, 20 g. CoI<sub>2</sub>, and 3 mL. AcOH heated 6 h. at 280-90° under 100 atm. CO gave phenanthrene, m. 100° (decomp.). Reactions, b<sub>2</sub> 95-123° (contg. C<sub>6</sub>H<sub>6</sub>): Cl<sub>2</sub> (b<sub>2</sub> 141H<sub>6</sub>, Cl<sub>2</sub> 141H<sub>4</sub>, and Cl<sub>2</sub> 141H<sub>2</sub>; isolated chromatog. on Al<sub>2</sub>O<sub>3</sub>, and not further characterized), b<sub>2</sub> 141-2° (probably dinaphthylbutyl ether, C<sub>28</sub>H<sub>30</sub>O), and b<sub>2</sub> 147-60° (naphthylbutanol, C<sub>14</sub>H<sub>16</sub>O). A mixt. of 720 g. 70% CH<sub>2</sub>:CHCH<sub>2</sub>OH and 870 g. 35% H<sub>2</sub>O<sub>2</sub> added to 100 mL. 0.5% OsO<sub>4</sub> pH adjusted with acid to 3-4), with simultaneous addn. of KOH to hold the pH at 3.5-3.75 gives glycerol in 90% yield. meso-Erythritol, prepd. similarly, m. 120° (from MeOH). Cl (100 g.) added to 140 g. 70% CH<sub>2</sub>:CHCH<sub>2</sub>MeOH in 860 mL. H<sub>2</sub>O at 10-20°, the mixt. stirred overnight, excess Cl swept out with air, 500 mL. 13% Na<sub>2</sub>CO<sub>3</sub> added, and the mixt. evapd. gives 100 g. HOCH<sub>2</sub>(CH(OH))<sub>2</sub>Me, b<sub>2</sub> 140-2°, di-Erythritol (dibenzal deriv., m. 218-19°) is prepd. similarly from HOCH<sub>2</sub>CH(OH)CHClCH<sub>2</sub>OH, b<sub>2</sub> 1.5 145° (decomp.); this, with 30% H<sub>2</sub>SO<sub>4</sub> gave 3,4-dihydroxy-XIIa, b<sub>2</sub> 121-2°. XXXVa chlorinated in CC<sub>14</sub> at 10° gives 3,4-dichloro-XIIa, b<sub>2</sub> 59-61°. HgO (135 g.) in 500 mL. H<sub>2</sub>O treated with 85 g. Cl with cooling, the mixt. filtered and distd. in vacuo, and 35 g. XXXVa added with cooling to the distillate (which contained HOCl) gives 36 g. 3-chloro-4-hydroxy-XIIa, b<sub>2</sub> 14

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102-3°; this (60 g.) added in vacuo to warm milk of lime and the vapors condensed and redistd. gives 24 g. 3,4-oxido-XIIa (XLVII), b. 143°, b14 45°. XLVII, ROH, and Al2O3 at 100° gave monoesters of 3,4-dihydroxy-XIIa (R and const. given): H, b13 163°; Me, b11 107°; Et, b12 112°; Bu, b15 134°; HO(CH2)4, b12 201°; Ph, b0.5 131°; PHCH2, b0.6 162°; 2,5-dimethyl-3-hydroxy-4-methoxy-XIIa, b10 102°. XLVII (86 g.) cooled and 400 g. 25% NH3 added gives 3-hydroxy-4-amino-XIIa, b11 142°, m. 78°.

Amino derivs. prepd. from XLVII and R1R2NH at 200° are (R1 and R2 given): octadecyl, H, b0.5 205°; Ph, H, m. 105° (from ligroine); Ph, Me, m. 63-4°, b2.5 175°; pyrrolidine, b17 153°. XXV bis(tetrahydro-3-furyl) ether, b2 145-50°, and 71 g. Cl added simultaneously in 15-20° during 2 h. to 2300 mL. H2O give 284 g. crude chlorohydrin which, added to 74 g. powd. Ca(OH)2 in 500 mL. H2O, gives 150 g. 2,3-oxido-1,4-butylene bis(tetrahydro-3-furyl) ether, b1 162-70°. XXV (665 g.), treated at 5-10° with 247 g. HCl gives 300 g. 3-chloro-XIIa, b17 70-5°. XLII (280 g.) and 9 g. powd. "Na dibutyl-naphthalenesulfonate" in 10 L. H2O treated at room temp. with 205 g. Cl dild. with air gives an unidentified tetrachlorobutane, b20 93-5°, and 287 g. CH2ClCHClCH(OH)CH2Cl(XLVIIa), 2 isomers, b1.2 74-6° and b1 103-4°. XLVIIa (142 g.) and 60 g. powd. Ca(OH)2 in 70 mL. H2O shaken 15 min. gives 70 g. of a mixt., b3 68-71°, of 1,4-dichloro-2,3-oxidobutane and 1,2-dichloro-3,4-oxidobutane. H2O2 (110 g. 40%) added to 125 g. XLII, 200 g. AcOH, and 0.04 g. OsO4 gives, after 20 h., 56 g. CH2Cl[CH(OH)CH2]2CH2Cl, m. 127°, this (80 g.) in 1 L. XIIa refluxed 3 h. with 60 g. NaOH gives 30 g. 1,2,3,4-dioxidobutane, b23 50-1°. XXV (200 g., 93%) in 1 L. AcOH chlorinated 5 h. at 0-10°, gives 280 g. (CHClCH2OH)2, b1 134-6°, m. 70°. (CHBrCH2OH)2, prep. similarly in CH2Cl2, m. 87° (from C6H6), b1.3-1.5 148-50°. HCl passed into 40 g. HOCH2CH(OH)CH2CH2OH and 8 g. AcOH at 80-100° until 300 g. has been absorbed gives 420 g. CH2ClCH(OH)CH2CH2Cl, b18 95-100°, 400 g. of this in 550 mL. H2O and 112 g. NaOH in 250 mL. H2O at 10-20° give 240 g. 1-chloro-3,4-oxidobutane, b45 63°, 190 g. of which heated to 120-30° with 90 g. powd. NaOH gives 100 g. butadiene monoxide, b. 68°. XXV (88 g.) and 89 g. anthracene in 3 h. at 250° under N gives 9,10-endo-(2',3'-butane-1',4'-diol)anthracene (XLVIII), m. 221° (from alc.); diacetate ester, m. 135° (from alc.-H2O). Similarly XXXVa gives, with anthracene, 9,10-endo-(3',4'-tetrahydrofuryl)anthracene, m. 180° (also obtained from XLVIII and 35% H2SO4 refluxed 7 h.), and, with 9,10-dichloroanthracene, 9,10-dichloro-9,10-endo-(3',4'-tetrahydrofuryl)anthracene, m. 165°. [MeCH(OH)CH]2 analogously gives 9,10-endo-(3',4'-hexane-2',5'-diol)anthracene, m. 213°. XIIa (10 g./h./L. catalyst) and NH3 (molar ratio 1:20, resp.) passed over Al2O3 at 300-20° gives 80-5% pyrrolidine (XLIX), b. 87-8° (N-Ac deriv., b. 112-14°, p-O2NC6H4CO deriv., m. 92-3°), 1-(3-butenyl)-XLIX, b. 152-4°, b14 64-5° [picrate, m. 105° (from alc.)], 1-(4-aminobutyl)-XLIX, b. 205° (picrate, m. 126° di-Bz deriv., b1 180°), 1,4-di(1-pyrrolidinyl)butane, b1 100° (picrate, m. 158°; dihydrate, m. 70°); N(CH2CH2CH2)2, b1 120°. HO(CH2)4OH

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comps. are prep. by conventional methods from XLIX and halo (usually Cl)

comps. (R = 1-pyrrolidinyl): (CH2CH2R)2, b22 146-52°; (RCO2CH2CH2)2, b1 190-200°, m. 61°; p-O2NC6H4R (LI), m. 166° (hydroxyl Ni-Cr2O3 at 70-80°, 200 atm. p-H2N analog (LII), b2 140°, m. 35°); p-O2NC6H4CH2R, hydrogenated to the p-NH2 analog, b3 129°, m. 51° (from ligroine); 1-(3-quinolyl)pyrrolidine, m. 96-8° (from ligroine); m-O2NC6H4SO2R; p-AcNHC6H4SO2R, m. 177° (from H2O); p-H2NC6H4SO2R, m. 168° (from H2O) (by hydrolysis of the preceding compd. with 5N H2SO4); 3,4-Cl2C6H3SO2R, m. 112° (from ligroine); 3,5,2-Cl2(HO)C6H2SO2R, m. 146° (from 70% alc.); 2,1,4,6-Me(HO)C6H2(SO2R)2, m. 153° (from ligroine); 3,4-Cl2C6H3SO2R, m. 126°. LIJ sulfate (80 g.) in 250 g. concd. H2SO4 and 115 mL. H2O treated at 110° with 72 g. Li, and 95 g. glycerin, H2O diatd. to raise the temp. to 140° and held there 6-7 h. gives 1-(6-quinolyl)-XLIX, b2 145-70°, m. 117° (from C6H6-ligroine). The sulfonilide (20 g.) from HO3SCl and 4,2-ClMeC6H3OH refluxed 2 h. with 40 g. XLIX give 2,5,4-ClMe(HO)C6H2SO2R (R=1-pyrrolidinyl, m. 126° (from ligroine)).

2-Mercaptobenzothiazole (334 g.) in 1500 mL. of H2O treated with 426 g. XLIX, then 5 L. 15% NaOCl gives 2-benzothiazolylsulfenpyrrolidide, C11H12N2S2 (LIJ), m. 178°. VIIJ (400 g., 30%) and 288 g. iso-PrCHO added with stirring and cooling to 284 g. XLIX and stirred several hrs. at 40-50° give 2,2-dimethyl-3-(1-pyrrolidinyl)propionaldehyde, b14 85°; this with NH3 and 150 atm. H at 150° over Ni gives 2,2-dimethyl-3-(1-pyrrolidinyl)propylamine, b0.5 55-7°. XLIX (200 g.) and 200 atm. H 12 h. at 200° over 40 g. Raney Co give 27 g. 1-Bu-XLIX, b. 150-4° (picrate, m. 125-7°), and a small amt. of (CH2CH2R)2 (R = 1-pyrrolidinyl), b3 138°; similar reaction in the presence of 200 g. NH3 gives 50 g. 2-Bu-XLIX, b. 154-6° (picrate, m. 122-3°), at 300° over Raney Ni. H2N(CH2)4NH2 and 1-H2N(CH2)4-XLIX, b10 89-119° (picrate, m. 125°), and 1,4-di(1-pyrrolidinyl)butane, b10 121-6° (picrate, m. 157°), are formed. IX (190 g. 50% aq. soln.), satd. with NH3 and passed during

3 h. over 250 mL. 5% ThO2 on Al2O3 over which 200 L./h. NH3 is circulated gives 61 g. unchanged IX and 30 g. pyrrole (LIV), b. 130°. A mixt. contg. 2/3 1-Me-LIV, b. 112-13°, and 1/3 2-Me-LIV, b. 147-8°, is refluxed in 10% yield from 1-Me-XLIX and 200 atm. H2O with N over a ZnO-CuO-Al2O3 catalyst. 2-Me-LIV is also prep. in 84% yield from 1-Me-LIV over clay chips at red heat; L dehydrogenated similarly gives 80% 1-Ph-LIV, m. 56-8°, rearranged as above to 2-Ph-LIV, m. 122-3°. XXXVa (180 g.), (or an equiv. amt. of XXV or HOCH2CH(OH)CH2CH2OH) and 228 g. PhNH2 over ThO2-Al2O3 at 300° give 1-phenyl-2,5-dihydropyrrole, b5 85-93°, m. 88° (from MeOH); this, 50 g., in 200 mL. C6H6 added to 90 g. PhNMeCHO and 110 g. POC13 in 100 mL. C6H6, held 2-3 h. at 0-10° and 12 h. at room temp., gives 4-(2,5-dihydro-1-pyrrolyl)benzaldehyde, m. 90-1° (from cyclohexane); semicarbazone, m. 270° (from alc.). XIIa (100 g., 94%) added to 662 g. 65% HNO3 and 4 g. NaNO2 at 25° gives 139 g. (CH2CO2H)2; its anhydride (LIVa) is prep. by passing this over Al2O3 or SiO2 at 275° and 130-60 mm. HO2CCHMeCH2CO2H is prep. similarly from 3-Me-XIIa; the anhydride, m. 30-5°, b2 105°. XXXVa (100 g.), passed at the rate of 6 g. and 240 L. air/h. over 100 mL. catalyst (prep. by heating 110 g. (NH4)2MoO4, 32 g. vanadic acid, and 40 g. TiO2 1 h. with 1 L. 4% (CO2H)2, adding 30 g. NH4Cl, then 500 g.

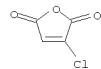
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similarly gives 85% XLIX and 6-8% high-boiling compds. contg. HN(CH2CHCH2)2, b. 155-60° (picrate, m. 248°). XLIX or HO(CH2)4OH and RNH2 over Al2O3 or ThO2-Al2O3 at 300-20° give the following 1-derivs. of XLIX (substituent given): Me, b. 79-81°; Et, b. 105-7°; Pr, b. 130-5°, b15 32-5°; Bu, b. 154-6°, b15 48-50°; n-C10H21, b1 114-16°; CH2CH2NH2, b10 55-7°; 1,2-di(1-pyrrolidinyl)ethane (formed with the preceding compd.), b9 98-100° (hydrate, m. 50°); Ph (L), b1 102-4°, m. 11° (L couples with diazo compds.; with HNO2 L gives green p-ON deriv., m. 128°); α-naphthyl, b0.4 137°; cyclohexyl, b. 213-15°; 2-pyridyl, b11 120-1°; 3-ethyl-6-methyl-2-pyridyl, b10 120-30°; 4-quinolyl, b0.8 158-60°. Analogously 222 g. HOCH2CH(OH)CH2CH2OH and 205 g. PhNH2 give 76 g. 1-phenylpyrrolidine. [MeCH(OH)CH2]2 gives 1-phenyl-2,5-dimethyl-XLIX, b0.6 80-6°; tetrahydropyran with NH3 gives piperidine and 1-(pentenyl)piperidine, b13 83-8°; PhNH2 gives 1-phenylpiperidine, b15 135-6°. L (27 g.) added at 10° to 50 g. PhNMeCHO and 69 g. POC13, the mixt. warmed several hrs. to 40-5°, and decompd. with ice gives 4-(1-pyrrolidinyl)benzaldehyde, m. 81° (from Et2O-petr. ether); semicarbazone, m. above 300°. Similarly L and COCl2 in CHCl3 at 5° give 4-(1-pyrrolidinyl)benzoyl chloride, subliming at 100-50°/0.1 mm.; hydrolysis gives the acid, m. 294-5° (from alc.). HOCH2CH(OH)CH2CH2OH (160 g.), 115 g. PhNH2, and 160 g. PhNH2.HCl heated

12 h. at 190° gives 50 g. 3-hydroxy-L, b1 138-44°; 1-m-tolyl-2-hydroxy-XLIX prep. similarly from m-MeC6H4NH2, b0.7-0.5 131-40°; 2-methyl-L, 100 g. from 172 g. XXXV, 230 g. PhNH2, and 30 g. fullers earth 12 h. at 270°, b13 125-35°. XLIX (93 g. 90%), 66 g. m-MeC6H4OH, 60 mL. concd. HCl, and 200 mL. H2O 12 h. at 160° give 50 g. 1-(m-hydroxy)-L, m. 128°; 1-β-naphthyl-XLIX, m. 92°, 250 g. from 216 g. β-naphthol, 200 g. XLIX, and 20 g. fullers earth 6 h. at 200°; 2,6-di(1-pyrrolidinyl)naphthalene, m. 240°, was prep. similarly, in 90 g. yield from 70 g. 2,6-dihydroxynaphthalene and 80 g. XLIX. Ethylene oxide (246 g.) passed at below 50° into 355 g. XLIX and warmed 1 h. at 50° gives 213 g. 1-(2-hydroxyethyl)-XLIX, b1 57-62°, 102 g. 1-[2-(2-hydroxyethoxy)ethyl]-XLIX, b1 92-4°, and 29 g. 2-(2-hydroxyethoxy)ethyl β-1-pyrrolidinylethyl ether, b1 120-4°. VIII (100 g. 30%), and 142 g. XLIX in 1 h. at 50° give 130 g. di(1-pyrrolidinyl) methane, b10 82-5° (1-Me-XLIX gives bis(2-methyl-1-pyrrolidinyl) methane, b1 57-8°). The following 1-substituted XLIX are prep. (substituent given): CH2CH2CO2Me, b0.5 66-8°; CH2CH2CN, b20 105-6° [this with 100 atm. H at 100° over Co gives amine, CH16N2, b10 76° (monopicrate, m. 145°); mono-p-aminobenzoyl deriv., m. 138°]; CHO, b20 112-14°, and a compd. contg. 2 mol XLIX and 1 mol CO (from XLIX and CO); CONH2, m. 218° (from H2O). CS2 (144 g.) and 213 g. XLIX treated with Me2SO4 give 100% N-pyrrolidinylidithiocarbonic acid Me ester, b9 155-8°, m. 90°. p-Toluenesulfonocyanate (197 g.), and 71 g. XLIX, react on mixing to give N-(1-pyrrolidinylcarbonyl)-p-toluenesulfamide, C12H16N2O3S, m. 214° (from alc.). XLIX (284 g.), and 142 g. (CH2ClCH2)2O heated 12 h. at 160° give 170 g. bis(2-(1-pyrrolidinyl)ethyl) ether, b22 166-72°. The following

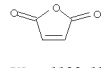
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pumice, evapg., and heating 2 h. at 300° gives 120-30 g. maleic acid (LV) and anhydride; 100 g. XIIa similarly gives 80-90 g. LV and anhydride. Cl (70-80 g.) passed into 100 g. LiVa at 150-80° gives 60 g. LV anhydride and 40 g. unchanged LiVa. Monochloro-LV anhydride is prep. from maleic anhydride (or LiVa), Cl, and FeCl3 at 160-80°, 100 g. LiVa, some FeCl3, and 240 g. Cl at 160-80° give 150 g. dichloromaleic anhydride. LiVa (600 g. 80%), treated at 190-200° with 3 g. NaOBz gives 260 g. γ-oxopimelic acid dilactone, b1 170°; 300 g. of this refluxed 60 h. with 600 mL. EtOH and 30 g. concd. H2SO4 give 305 g. diester of γ-oxo-pimelic acid, b12 165°. Hydrogenation of the dilactone in alk. soln. at 160°, 200 atm. gives 90% γ-hydroxypimelic acid; 500 g. of the dilactone, and 350 g. NaHCO3 in 1500 mL. H2O hydrogenated over 35 g. Raney Ni and 35 g. Cu chromite at 280°, 200 atm. give 220 g. pimelic acid.

IT 96-02-6P, Maleic anhydride, chloro- 108-31-6P, Maleic anhydride 1122-17-4P, Maleic anhydride, dichloro- RL: PREP (Preparation) (preparation of)

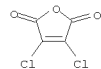
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CN 2,5-Furandione, 3-chloro- (CA INDEX NAME)



RN 108-31-6 CAPLUS  
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RN 1122-17-4 CAPLUS  
CN 2,5-Furandione, 3,4-dichloro- (CA INDEX NAME)





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